(160)

Manned Spacecraft Center Internal Note No. MSC IN 65-EP1

# EFFECTS OF EXPOSURE OF VARIOUS ELECTRICAL CONDUCTORS TO NITROGEN TETROXIDE, AEROZINE - 50 AND POTASSIUM HYDROXIDE


Prepared by: Herschel Jamison
Chief Chemist, Thermochemical Test Branch
Materials Laboratory

Approved by: Jesse C. Jones
JESSE C. JONES
Chief, Thermochemical Test Branch

Approved by: Joseph G. Thibodaux, Jr.
JOSEPH G. THIBODAUX, Jr.
Chief, Propulsion and Power Division



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Manned Spacecraft Center

Houston, Texas

January 15, 1965

TABLE OF CONTENTS

# EFFECTS OF EXPOSURE OF VARIOUS ELECTRICAL CONDUCTORS

# TO

# NITROGEN TETROXIDE, AEROZINE - 50, AND POTASSIUM HYDROXIDE

## INTRODUCTION

The purpose of this test program was to determine the effects on the serviceability of various insulated electrical wire conductors when exposed to propellants and chemicals utilized in the propulsion and power systems of the Apollo Spacecraft. Eleven (11) electrical wire conductors with various types of insulation were exposed to liquid nitrogen tetroxide, aerozine - 50 (50-50 blend of unsymmetrical dimethyl hydrazine and hydrazine) and molten potassium hydroxide.

The test program was conducted by the Thermochemical Test Branch Materials Laboratory for the Reliability and Flight Assurance Division. The program was conducted at the interim Thermochemical Test Branch Chemical Laboratory located at Ellington Air Force Base, Texas, during the period from February 4, 1964, to February 11, 1964.

## DESCRIPTION OF TEST SPECIMENS

The conductors in the eleven (11) test specimens were made of copper, some of which were tin or silver plated. The conductor sizes were all 20 gauge (American Wire Gauge - AWG) with the exception of one wire which was 22 AWG. All conductors were 19 strand, 32 wires per strand except the Surprenant Thermorad TRT which contained 7 strands, 28 wires per strand. Various insulating materials were used on the test specimens with wall diameters varying between .006 inches and .013 inches (see Table 1, page 12). The various insulating materials used are as follows:

a. IMP -- Irradiated Modified Polyolefin. This material is a common plastic material which has had its basic molecular structure modified by crosslinkage under the influence of irradiation. This modification presumably improves the dielectric characteristics of the material.

b. FEP -- Fluorinated Ethylene Propylene. This material is a plastic copolymer of polyethylene and polypropylene which has been combined with fluorine to give a fluorocarbon material related to tetrafluoroethylene (teflon). The fluorine imparts inertness to oxidation; the ethylene imparts flexibility; and the propylene, toughness. This compound has a truer melting point than teflon; thus, it is easier to extrude, and it covers the wire more uniformly.

c. TFE -- Tetrafluoroethylene. This is an extremely inert material which has excellent chemical resistance, heat tolerance, and dielectric characteristics. This material is essentially polyethylene which has some fluorine atoms added to, and substituted in, the molecular chain. This polymer has gained wide acceptance in many fields requiring a substance with good dielectric properties, toughness, heat resistance, and chemical resistance. It is commonly called teflon.

d. FEP/ML -- Fluorinated Ethylene Propylene/Polyimide Lacquer. This is a fluorinated ethylene propylene material coated with ML polyimide lacquer. The ML coating, though less than 1 mil thick, gives good cold flow abrasive resistance and improvement in the mechanical properties. The melting point for this material is 200°C, which establishes its ultimate extended use temperature.

e. TFE/ML -- Tetrafluoroethylene/Polyimide Lacquer. This material is very similar to the FEP/ML except that its extended use temperature may run as high as 260°C.

f. Vinylidene Fluoride. This is a fluorinated vinyl polymer with good impact and abrasive qualities. This material has a definite drawback in that its dialectric strength is not as good as TFE. Brittleness at low temperature is also a problem with this material.

g. "H" Film/FEP. This is a polyimide film stock of the nylon family. It is a highly abrasive resistant polymer. Fluorinated ethylene propylene is used as a bonding material to hold the "H" film together.

h. "H" Film/TFE. This is a material with properties similar to "H" Film/FEP except that the bonding agent is TFE.

For further details on these test specimens, refer to Table 1.

## DESCRIPTION OF CHEMICAL ENVIRONMENTS

Aerozine - 50. This is a rocket engine fuel blend procured in accordance with specification MIL-P-27402 (USAF). Aerozine - 50 is a very toxic alkaline solution which is highly reactive, especially to those substances which are oxidative. Many substances which are not normally considered oxidative are reactive to Aerozine - 50 due to its extremely reduced state. It is also an excellent solvent for many substances. Under proper conditions it will even attack ceramic materials which are considered to be quite inert.

Nitrogen Tetroxide. This is a rocket engine oxidizer procured in accordance with specification MIL-D-26539 (USAF). Structurally, nitrogen tetroxide in the liquid state is a dimer made up of two molecules of nitrogen dioxide, which is the anhydride of nitric acid. This means that in the presence of moisture, the extremely reactive chemical, nitric acid, is formed. This acid exists in a very highly oxidative state and is normally very corrosive to organic polymers. Careful selection, then, must be made in choosing a wire insulation which will resist this attack. Generally speaking, materials which resist the reducing attack of hydrazine are susceptible to the oxidizing attack of nitrogen tetroxide.

Potassium Hydroxide. This material is used as an electrodyte in the Apollo Spacecraft fuel cells and is in the molten state during active operation of the cells. It is an extremely strong base and will attack such materials as glass and ceramics which are normally inert to all common acids except hydrofluoric. In the molten state, this chemical is extremely reactive to all known wire insulation materials. This destructive power is more attributed to the heat involved, in most cases, rather than to chemical activity.

## DESCRIPTION OF TEST APPARATUS

Samples of the eleven (11) test specimens were exposed to liquid nitrogen tetroxide, a 50-50 mixture of UDMH and hydrazine (Aerozine-50), and molten potassium hydroxide. The tests were accomplished within a type 304 stainless steel test chamber approximately eight (8) inches in diameter and 20 inches in length. The test chamber incorporated a teflon gasket to seal the lid, and a safety relief valve was provided to protect the vessel against over pressurization (see Figure 1). In order to mount the test specimens in suitable positions, a stainless steel clamp-type frame was devised (see Figure 2). The test specimens were protected from physical damage by strips of teflon sheets held in place by metal straps. Following the mounting of the test specimens in proper position (separated by approximately 1/4 inch), the frame was then inserted inside the chamber where it rested on the bottom of the vessel. The chemicals were then introduced.

## TEST PROCEDURE

Prior to exposure to the test environment, a sample of each wire specimen was subjected to a dielectric test utilizing a Multi-Amp 4D-3A-1, 0-10 kilovolt direct current (KVDC) tester. All samples with 0.008 inch or greater insulation wall thickness were tested at 2.0 KVDC, while those with less than 0.008 inch wall thickness were tested at 1.5 KVDC. The current leakage was measured with a 50 microamp meter. All dielectric tests were accomplished with the test samples coiled in 2 to 3 inch diameter coils submerged in water for a period of 30 seconds. See Table 2 for results of the dielectric tests.

Following the initial dielectric tests, the test specimens were mounted on the stainless steel frame, placed in the test chamber, and exposed to the chemical environment for a period of seven days. After the seven days had expired, all the specimens were thoroughly washed with water, photographed, and subsequently tested again for change in dielectric characteristics. Microscopic inspection of the test specimens was also conducted.

## RESULTS AND DISCUSSION

Prior to exposure of the wire samples to the test environment dielectric tests were accomplished in accordance with the techniques described under the paragraph: Test Procedures. No electrical leakage was found in any of the samples during the initial dielectric test. Notation was also made of the specimen insulation color in order to recognize chemical reaction by change of color during the test. Microscopic examination was also accomplished on the cross-sectional area of each wire to determine if exposure would cause separation of the insulation from the bare conductor with subsequent attack on the wire itself.

Test results are indicated below for each sample and each environment. Except in seven cases of very obvious destruction to the insulation, it was not possible through visual and microscopic inspection to accurately assess the deleterious effects of the various chemicals on the conductor samples. For this reason it is felt that conclusions as to the resistivity of the various samples to attack by the test environments should be based on the dielectric test results which are tabulated in Table 2. It is also considered highly **desirable** that additional testing be accomplished before conductor selections are made. This additional testing should include other types of tests such as flexibility and brittleness to determine the overall capability of the conductor to withstand the complete spacecraft environment.

The following is a summary of observations of each test sample following exposure to the three (3) environments:

### Nitrogen Tetroxide

Sample 1-69 (FMP): This sample was originally green. It was somewhat bleached following the test with corrosion showing on the bare wire.

Sample 2-73 (FEP): This specimen was originally white in color. Following the test, no change in color was noted. Considerable green colored corrosion products were discovered on the bare wire indicative of copper nitrate as a result of chemical reaction between the copper conductor and the nitrogen tetroxide.

Sample 3-74 (IMP): Initially this sample was green in color which bleached out during the test. Corrosion was observed in the bare wire.

Sample 4-75 (TFE): This sample was white prior to the test as well as after. Copper nitrate corrosion also showed on the bare wires.

Sample 5-76 (IMP): This specimen remained off-white in color throughout the test. Corrosion was noted on the bare wires.

Sample 6-77 (FEP/ML): This sample was initially yellow in color. During the test, the yellow color was changed to green which showed up under the insulation all along the wire indicating severe corrosion of the wire and chemical instability of the insulation.

Sample 7-78 (TFE): Originally this sample was white; following the test, the color had changed to off-white. Evidence of wire corrosion was also present.

Sample 8-89 (TFE/ML): The original yellow color of this sample was retained through the test. The yellow color was due to the ML coating. This coating became very brittle during the test; thus, sharp bending produced cracks indicating a failure of the insulation.

Sample 9-90 (H Film): This sample was originally gold in color. The final color was a bright brown which indicates chemical reaction in the insulation. Some corrosion was noted in the bare wire at the exposed ends.

Sample 10-93 (Vinylidene fluoride): This specimen was originally green in color. This color was almost entirely bleached out in the test. The bare ends on the wire also showed typical copper nitrate corrosion.

Sample 11-99 (H Film/TFE): This sample held its white color throughout the test. The bare ends on the wire were corroded. Results of exposure of the wire samples to nitrogen tetroxide are summarized in Table 3.

## Aerozine - 50

Sample 1-69 (FMP): This sample was originally green in color but was bleached out by the test. No corrosion was observed on the wire.

Sample 2-73 (FEP): This sample remained white through the test. No corrosion was apparent.

Sample 3-74 (IMP): This sample showed no appreciable visible change.

Sample 4-75 (TFE): This sample retained its white color and no corrosion was apparent.

Sample 5-76 (IMP): This sample showed no notable change.

Sample 6-77 (FEP/ML): This sample was originally yellow in color but turned off-white during the test. Minor corrosion was present on the bare wire.

Sample 7-78 (TFE): This sample was originally white in color but turned to a dark, streaked brown during the test.

Sample 8-89 (TFE/ML): This sample was bleached white from an original yellow color. No corrosion was apparent on the bare wire.

Sample 9-90 (H Film): This sample was originally gold in color, but changed to a bright yellow during testing. The insulation was severly attacked; it showed extensive delamination of the outer wrapping. Fine yellow powder was prevalent between the wrappings. No corrosion was evidenced on the bare wire.

Sample 10-93 (Vinylidene fluoride): This sample was orginally green in color but changed to a dirty yellow during test. Some corrosion was apparent on the bare wire.

Sample 11-99 (H Film/TFE): This sample remained white during the test. No corrosion of the wire was apparent. Results of exposure of the wire samples to Aerozine-50 are summarized in Table 4.

## Potassium Hydroxide

Samples listed below had their insulation completely disintegrated with only the bare wires remaining:

No. 6 (FEP/ML)

No. 9 (H Film)

No. 10 (Vinylidene fluoride)

No. 11 (H Film/TFE)

The balance of the samples, 1, 2, 3, 4, 5, 7, and 8 showed somewhat less destruction to the insulation. In every case, however, the wire insulation showed severe shrinkage and enough destruction to indicate failure to the wires. Destruction was so great to all the samples that the dielectric test was cancelled for it was apparent that the wires would fail.

It should be noted that the test in potassium hydroxide is an extremely severe test due to the heat involved. The temperature of 460$^{\circ}$F was chosen because this is the nominal operating temperature of the potassium hydroxide in the Pratt and Whitney fuel cell. The results of this phase of the test should not be used to evaluate wiring which is intended for use in anything except the fuel cell itself. The likelihood of molten potassium hydroxide contacting other spacecraft wiring is so remote as to discount it. Results of exposure of the wire samples to molten potassium hydroxide are summarized in Table 5.

## CONCLUSIONS

1. Tetrafluoroethylene (Teflon) coated conductors as a group withstand exposure to nitrogen tetroxide and Aerozine-50 more adequately than conductors coated with other materials.

2. Bleaching of the color from the conductor insulation was not an adequate parameter for failure analysis **determination.**

3. Aerozine-50 was more destructive, as a whole, to the eleven types of wire tested than was the nitrogen tetroxide as determined by the dielectric tests.

   NOTE: From visual observations, however, one would conclude the opposite inasmuch as physical changes such as color and corrosion were more pronounced in the nitrogen tetroxide exposed samples.

4. The most reliable method of assessing electrical wiring environmental test results is by dielectric tests.

5. The following conductor insulating materials appeared to satisfactorily withstand the exposure to both nitrogen tetroxide and Aerozine-50:

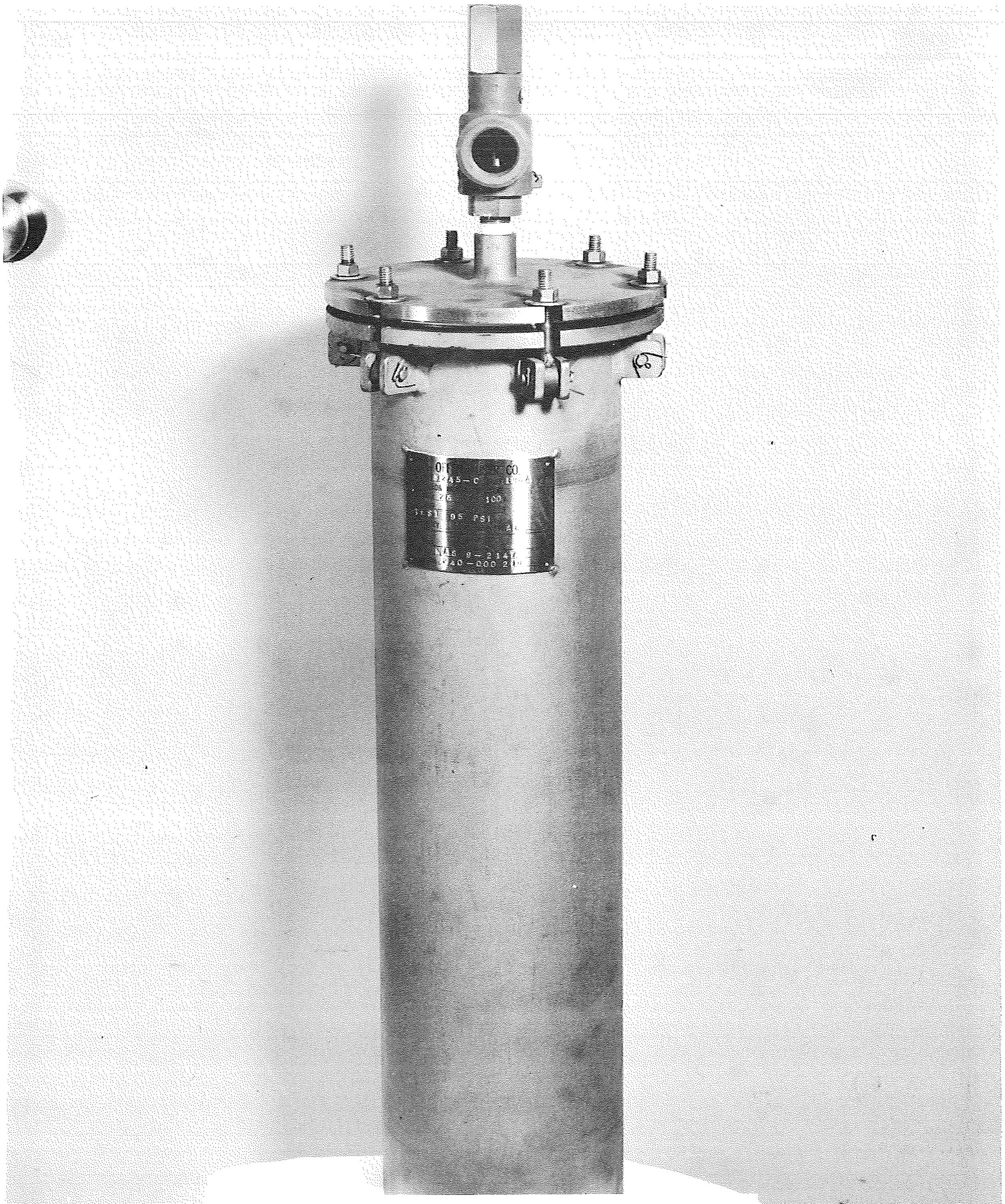
   a. Fluorinated Ethylene Propylene (Sample 2-73)

   b. Tetrafluorethylene (Sample 4-75 and 7-78)

   c. Irradiated Modified Polyolefin (Sample 5-76)

6. It is recommended that further testing be accomplished and that additional tests such as flexibility and brittleness be included before final decisions are made on electrical conductor selections.

7. It is recommended that for future tests wire specimens be properly potted at the ends in order to prevent liquids and gases from entering the cavities between the insulation and the wires.



NASA
S-64-16942

FIGURE 1
TYPE 304 STAINLESS STEEL
TEST CHAMBER

NASA
S-64-16940

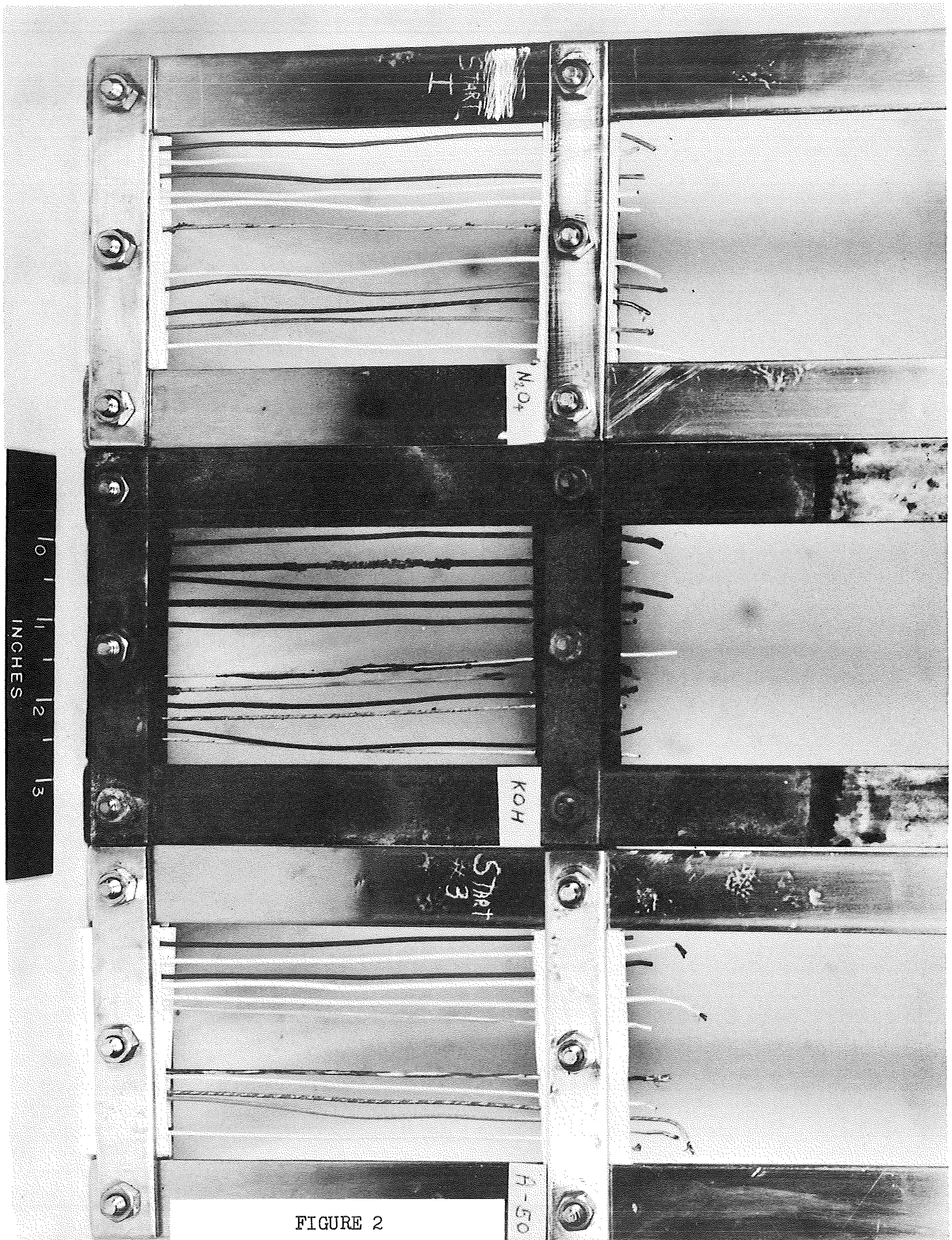



FIGURE 2
TEST SPECIMEN HOLDING FRAME

## TEST SPECIMEN SPECIFICATIONS

### TABLE I

| SAMPLE | INSUL. MTL. | O.D. INCHES | INSUL. WALL THICKNESS | INSUL. | GAUGE | CONDUCTOR | PLATING | STRANDS | SUPPLIER |
|---|---|---|---|---|---|---|---|---|---|
| 1(69) | IMP | .059 | .010 | green | 20 | copper | tin | 19/32 | McDonnell |
| 2(73) | FEP | .060 | .010 | white | 20 | copper | silver | 7/28 | Hughes |
| 3(74) | IMP | .058 | .009 | green | 20 | copper | tin | 19/32 | NASA IESD |
| 4(75) | TFE | .067 | .013 | white | 20 | copper | silver | 19/32 | Hughes |
| 5(76) | IMP | .058 | .009 | off-white | 20 | copper | tin | | Space Tech Labs |
| 6(77) | FEP/ML | .052 | .006 | yellow | 20 | copper | silver | | Hughes |
| 7(78) | TFE | .060 | .010 | White | 20 | copper | silver | 19/32 | Chance Vought |
| 8(89) | TFE/ML | .046 | .077 | yellow | 22 | copper | silver | | Surprenant |
| 9(90) | H Film | .052 | .006 | gold | 20 | copper | | 19/32 | Martin-Orlando |
| 10(93) | vinylidene fluoride | .053 | .007 | green | 20 | copper | | 19/32 | Martin-Orlando |
| 11(99) | H Film/ TFE | .065 | .008 | white | 20 | copper | silver | 19/32 | Hughes |

LEAKAGE

| SAMPLE NO. | D.C. VOLTAGE | BEFORE EXPOSURE | AFTER EXPOSURE TO $N_2O_4$ | AFTER EXPOSURE TO A-50 |
|---|---|---|---|---|
| 1-69 | 2,000 | None | No leak indicated at 2000 VDC | 2-3 megohms at 1.5 VDC |
| 2-73 | 2,000 | None | No leak indicated at 2000 VDC | No leak indicated at 2000 VDC |
| 3-74 | 2,000 | None | 400 to 1000 megohms at 2000 VDC | 60 megohms at 100 VDC |
| 4-75 | 2,000 | None | No leak indicated at 2000 VDC | No leak indicated at 1500 VDC. |
| 5-76 | 2,000 | None | No leak indicated at 2000 VDC | No leak indicated at 2000 VDC |
| 6-77 | 1,500 | None | No leak indicated at 2000 VDC | 3 mehomns at 500 VDC-Breakdown at 500 VDC |
| 7-78 | 2,000 | None | No leak indicated at 2000 VDC | No leak indicated at 2000 VDC |
| 8-89 | 2,000 | None | No leak indicated at 2000 VDC | No leak indicated at 2000 VDC |
| 9-90 | 1,500 | None | Direct short--broke down at 400 VDC | Direct Short |
| 10-93 | 1,500 | None | 4000 megohms | 40 megohms at 2000 VDC |
| 11-99 | 2,000 | None | 40 megohms at 500 VDC | 3 megohms at 222 VDC-insul. crumbled |

TABLE 2

CONDUCTOR DIELECTRIC TEST RESULTS

| | | INSULATION COLOR | | |
|---|---|---|---|---|
| Sample | Insulation Material | Before Test | After Test | Remarks |
| 1-69 | Irradiated modified polyolefin | Green | Pale Green | The color was bleached out. Corrosion showed on the bare wire. |
| 2-73 | Fluorinated ethylene propylene | White | White | No color change. Green corrosion products showed on the bare wire. |
| 3-74 | Irradiated modified polyolefin | Green | Pale Green | The color was bleached out. Corrosion products showed on the bare wire. |
| 4-75 | Tetrafluoroethylene | White | White | Green corrosion products showed on the bare wire. |
| 5-76 | Irradiated modified polyolefin | Off-White | Off-White | No color change. Corrosion showed on the bare wire. |
| 6-77 | Fluorinated ethylene propylene | Yellow | Green | Color changed from yellow to green. The green showed up primarily under the insulation next to the bare wire. |
| 7-78 | Tetrafluoroethylene | White | Off-White | There was a slight color change. Corrosion products showed on the bare wire. |
| 8-89 | Tetrafluoroethylene polyimide lacquer | Yellow | White | The color was changed from yellow to white. Corrosion was noted on the bare wire. |
| 9-90 | H Film fluorinated ethylene propylene | Gold | Bright Yellow | Slight color change was noted. Fine yellow powder was noted in the wrappings. Corrosion showed on the bare wire. |
| 10-93 | Vinylidene fluoride | Green | Dirty Yellow | Some color change was noted. Some corrosion was noted on the bare wire. |
| 11-99 | H Film tetrafluoroethylene | White | White | No color change was noted. Corrosion was noted on the bare wire. |

TABLE 3

NITROGEN TETROXIDE EXPOSURE TEST RESULTS

| Sample | Insulated Material | INSULATION COLOR Before Test | After Test | Remarks |
|---|---|---|---|---|
| 1-69 | Irradiated modified polyolefin | Green | Pale Green | Color was bleached. No corrosion was noted on the wire. |
| 2-73 | Fluorinated ethylene propylene | White | White | No color change. No corrosion was noted. |
| 3-74 | Irradiated modified polyolefin | Green | Green | No color change. No corrosion was noted. |
| 4-75 | Tetrafluoroethylene | White | White | No color change. No corrosion was noted. |
| 5-76 | Irradiated modified polyolefin | Off-White | Off-White | No color change. No corrosion was noted. |
| 6-77 | Fluorinated ethylene propylene polyimide lacquer | Yellow | Off-White | Considerable color change was noted. No corrosion was noted. |
| 7-78 | Tetrafluoroethylene | White | Streaked brown | Considerable color change was noted. No corrosion was noted. |
| 8-89 | Tetrafluoroethylene polyimide lacquer. | Yellow | White | Some color change was noted. No corrosion was noted. |
| 9-90 | H Film fluorinated ethylene propylene | Gold | Bright yellow | Some color change was noted. No corrosion was noted on the bare wire. |
| 10-93 | Vinylidene fluoride | Green | Dirty yellow | Some color change was noted. No corrosion was noted. |
| 11-99 | H Film tetrafluoroethylene | White | White | No color change was noted. No corrosion of the wire was noted. |

TABLE 4

AEROZINE-50 EXPOSURE TEST RESULTS

| Sample | Insulation Material | Remarks |
|---|---|---|
| 1-69 | Irradiated modified polyolefin | Insulation was approximately 50% disintegrated |
| 2-73 | Fluorinated ethylene propylene | Insulation was approximately 50% disintegrated |
| 3-74 | Irradiated modified polyolefin | Insulation was approximately 50% disintegrated |
| 4-75 | Tetrafluoroethylene | Insulation was approximately 50% disintegrated |
| 5-76 | Irradiated modified polyolefin | Insulation was approximately 50% disintegrated |
| 6-77 | Fluorinated ethylene propylene polyimide lacquer | Insulation was completely disintegrated |
| 7-78 | Tetrafluoroethylene | Insulation was approximately 50% disintegrated |
| 8-89 | Tetrafluoroethylene polyimide lacquer | Insulation was approximately 50% disintegrated |
| 9-90 | H Film fluorinated ethylene propylene | Insulation was completely disintegrated |
| 10-93 | Vinylidene fluoride | Insulation was completely disintegrated |
| 11-99 | H Film tetrafluoroethylene | Insulation was completely disintegrated |

TABLE 5

POTASSIUM HYDROXIDE EXPOSURE TEST RESULTS